Petroleum Distilling Equipment II

Tower Distillation

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TOWER DISTILLATION

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In order to become acquainted with the working of a distil-
ling-tower, we must learn the main processes of distillation.

Distillation begins with vaporization, that is, the process
in which the liquid changes its state and becomes vapor. This
process absorbs heat energy. If a carbon-hydrogen mixture is heated
under constant pressure, the heat energy will be used partly to cause
the above mentioned phase-change and partly to raise the temperature
of the liquid. With the rise of temperature the individual com-
ponents of the liquid strain to change from a liquid to a vapor,
and this effort is then expressed in the rise of their vapor pres-
sure. In the vapor phase all components are found that are present
in the liquid phase but, with the rise of temperature, the vapor
pressure of components having a lower boiling point rises faster
than that of those having a higher one. Consequently, the vapor
phase will be richer in components with a lower boiling point than
in those with a higher one.

The law of behaviour of gases, or vapors was summarized by
Dalton as follows: in a gas mixture each gas component has the
same pressure it would have if it were taking up alone the whole
space at its disposal. At the same time, the pressure of the
whole system will be equal to the total of the partial pressures
of the gas components, as expressed in the formula

$$p = \text{partial pressure} = \pi \cdot y$$

where "π" is the total pressure and "y" is the capacity or molecular
fraction of the material.

- 1 -

Just as all gas components have a partial pressure, all components of the liquid also have a partial vapor pressure. The partial vapor pressure depends on the concentration of the component in the liquid and on the vapor pressure of the pure component.

If P is the vapor pressure of the pure component at a given temperature, and x is the mole fraction of the component in the liquid, then

p = partial vapor pressure of the liquid = P.x

Related to the total pressure:

$$\frac{p}{\pi} = \frac{P}{\pi} \cdot x$$

In a balance system the partial vapor pressure of the component is equal to the partial vapor pressure of the component in the liquid. Accordingly, $p/\pi = y$, that is the mole fraction of the vapor component, and

$$y = \frac{p}{\pi} = \frac{P}{\pi} \cdot x$$

which formula expresses Raoult's law.

In case of two components the formula will be:

$$\pi \cdot y_1 = P_1 x_1 \quad \text{and} \quad \pi \cdot y_2 = P_2 x_2 ,$$

added

$$\pi (y_1 + y_2) = P_1 x_1 + P_2 x_2$$

but

$$x_2 = 1 - x_1 \quad \text{and} \quad y_1 + y_2 = 1$$

$$x_1 = \frac{\pi - P_2}{P_1 - P_2} \quad \text{and} \quad x_2 = \frac{\pi - P_1}{P_2 - P_1}$$

$$y_1 = \frac{P_1}{\pi} x_1 \quad \text{and} \quad y_2 = \frac{P_2}{\pi} x_2$$

- 2 -

The data for hydrocarbons containing few carbon atoms necessary for the use of above formula, are available today in the indexes of every textbook. Applied to the separation of normal pentane and hexane, the above formula shows the following numerical formulation:

Under atmospheric pressure, at 49 degrees Centigrade (120 degrees Fahrenheit:

the vapor pressure of C_5H_{12} is 1160 millimeters, and

the vapor pressure of C_6H_{14} is 396 millimeters;

substituted in the equation

$$x_1 = \frac{\pi - P_2}{P_1 - P_2} \qquad \text{and} \qquad y = \frac{P_1}{\pi} \, x_1$$

$$x_1 \text{ for } C_5 \quad H_{12} = \frac{760 - 396}{1160 - 396} = 0.477$$

$$y_1 \text{ for } C_5 \quad H_{12} = \frac{1160}{760} \cdot 0.477 = 0.73$$

Examining on the curve the balance of a pentane mixture of 0.3 mole fraction, we shall see that, in the vapor phase, the pentane content is of 0.546 mole fraction. However, only a small quantity of this vapor could be produced since, together with vapor production, the composition of the liquid part changes and its pentane content diminishes. When heating the liquid mixture, the pentane content of the liquid decreases step by step and, finally, in case of total evaporation, the total vapor quantity of the compound is equal to that of the original liquid.

It was necessary to deal with the above mentioned principles in order to get acquainted with the theory of fractionation.

For, when condensing the above-mentioned vapor of a 0.546 mole fraction, and then vaporizing it anew, the vapor thus produced will contain a pentane of 0.782 mole concentration. Repeating this process, we arrive at point C on the diagram which already has a 0.92 mole fraction pentane. At step D the pentane rises to 0.975 mole in the vapor stage.

The theory explained above is practically applied in the so-called fractionating tower. The vapor mixture, entering the tower and proceeding upward in it, mixes with the condensed vapors which are already there. As a result of mixing, an exchange of material and heat takes place, i.e. a theoretically balanced state forms between liquid and vapor. The vapor components having a higher boiling point, that is a lower vapor pressure, become condensed and thus change into the liquid phase while proceeding upward in the tower, as a result of diminishing temperatures. Accordingly, vapors proceeding upward will be richer in components having a lower boiling point.

The purpose of the interior equipment of the distilling tower, of the "plates" is to cause the ascending vapors and descending condensates to mix with each other and thus to cause a balance between vapor and liquid. The descending liquid stream, as opposed to the ascending vapors, is called "inside reflux" in the tower.

An inverse relationship exists between the quantity of reflux and the balance states, i.e. the number of plates, to achieve a sharpness in separation. In the case of an infinite number of plates, a minimum of reflux is necessary. The same is true in the

- 4 -

other extreme case: a minimum number of plates may be used if an infinite quantity of reflux is employed. Both cases are, of course, purely theoretical; in practice economic necessities regulate the needed ratio between reflux and number of plates.

During the distilling process, the balance states on individual plants can be figured out in advance. To establish the proper number of plates and the quantity of reflux, there are mathematical and graphic solutions available. These, however, can be used with perfect exactness only in the case of a few components with a low number of atoms. In the case of mineral oil distillation we are working with a system containing many unknown components. Consequently, in these cases, mathematical calculations are always only approximate. Therefore, we do not want to deal with calculations in any more detail since their precision is only approximate anyway. This is also the reason why planning in the mineral oil industry is nearly completely based on empirical data.

The first step in planning is the determination of the temperature of the material when it leaves the furnace. This temperature depends, of course, on how high a percentage of the material we want to vaporize. In case of atmospheric distilling towers, the petroleum generally is heated to the point when the gas oil vaporizes. However, there have been and still are built towers that distill 90 to 95 percent of the material. The furnace-exit temperature of these towers is, of course, substantially higher but whether they are economical is a matter of argument. By the use of these towers, all the petroleum, that is, in the case of the petroleum of Lispe, all its gasoline, kerosene, and

- 5 -

gas oil parts, constituting 75 percent of the total, should be heated about 50 degrees higher.

The calculation of furnace-exit, i.e. tower-entrance temperature is based on ASTM or Engler (laboratory) distillation, using at the same time the principle worked out by Piromov and Beiswenger. ASTM or Engler distillation is based on partial vaporization. Since at tower-entrance temperature a balance vaporization occurs, the conversion becomes necessary.

The vaporous parts of the hot hydrocarbon mixture, after entering the distilling tower, proceed bubbling upward through column plates. The liquid parts flow downward, to the bottom of the tower. According to experience, if the vapor phase contains about 10 percent of a hydrocarbon having a higher boiling point, the liquid phase will also contain about 10 percent light product.

The separation of products in the tower is effected on the theory of the above-mentioned fractionation. Modern distilling towers, with very few exceptions, are equipped with bubble-capped plates. However, there are also towers filled with simple perforated plates. The use of Raschig rings is very rare today; their only use is to clean the oil of traces of solutions. There is another reason why it is not desirable to fill larger towers with Raschig rings. This is because they cause the appearance of channels of vapor and liquid that prevent the meeting of the two phases on a larger surface and, consequently, the effectiveness of the tower deteriorates.

- 6 -

The effectiveness of bubble-cap plates is usually 80 percent
and that of perforated plates between 20 and 40 percent. (Effective-
ness means the quotient of the theoretical number of plates divided
by the number of plates needed to achieve the desired effect, multi-
plied by 100.)

In the case of petroleum distillation, the balance states
necessary for separation of individual fractions, i.e. the number
of plates in the tower, are planned on the basis of empirical data.
The following table shows the number of plates needed for the
separation of individual fractions by the towers, blowing in steam:

Light gasoline - heavy gasoline	5-6 plates
Heavy gasoline - kerosene	4-5 plates
Kerosene - gas oil	4-5 plates
Gas oil - light paraffin distillate	3-5 plates
Original input -- gas oil	2-3 plates
"Pacura" - vaporizing (perforated) plates	4-5 plates

Notwithstanding their lower effectiveness, the perforated
plates are often used, especially in cases when the material to
be distilled contains mechanical impurities or suspended solid
parts. This is the reason why perforated plates are considered
as original input by petroleum distilling towers.

The perforated plates are also more advantageous if carbon
formation appear. In the case of vacuum towers they are also often
used because their resistance to steam currents is lower than that
of bubble-cap plates and therefore, even in the lower part of the
tower, a better vacuum can be achieved. Perforated plates are also
built when a hot product is to be cooled by injection of cold water.

- 7 -

In this case plates are exclusively used for mixing purposes.

The use of bubble-cap plates is the most widespread today. The cross-section of such a tray is shown in the following diagram. [See end of text]

The vapors, proceeding from upwards, bubble through the tower under the bubble caps, then through the holes in the bubble caps and, finally, through the liquid. The condensed distillates flow downward from plate to plate, flooding the whole expanse of the plate where they contact the vapors which are streaming upwards. There are various bubble-cap forms but this does not influence the effectiveness of the plate, and, consequently, there is no final form which can be called better than others. There are bubble caps which are round, elongated, or angular, with open or closed teeth, and often with teeth bent outward. The material of the bubble caps usually is pressed steel or cast iron. The plate itself is made also mostly of these two materials. Cast iron is often used because it offers better resistance to corrosion. However, corrosion does not appear normally but at the meeting place of vapor and liquid; therefore the better ability of cast iron to resist corrosion cannot be used extensively.

In small towers the cast iron plates are usually made in one piece; in larger towers they are made of more pieces. In this latter case the edge of the individual pieces should be sealed carefully to prevent the liquid from flowing through onto the plate below. The material used for condensation usually is asbestos cord which should be fixed with an iron rod for, in practice, the pressure forming at the bottom of the tower sometimes blows out the condensate stopping

production in order to repair the loosened condensing cords.

When installing the pipes which conduct the condensate down, the main point is that the liquid should be spread evenly over the whole expanse of the plate. The arrangements used for different tower sizes is shown in the following diagram: [See end of text]

At great diameters small dams are absolutely necessary, that is, some caps should be installed to serve as dams.

The bubble-cap solution, though very widespread, might still not be the final word in the problem. New patents are constantly being registered, all of which attempt to find another solution to the problem. The reason is partly that the bubble-cap construction is somewhat expensive and partly that in towers with a large diameter it is very difficult to make the liquid spread evenly and, as a consequence, the effectiveness of the plate diminishes.

Diagram No 4 shows a proposed patent registered in a foreign country. [See end of text]

In the past few years there was only one proposal which competed seriously with the bubble-cap plate. This is the so-called cascade-insert shown in the following diagram. [See end of text]

The vapor proceeds upward in the direction of the arrows and the liquid, leaving the pipe shown on the left side of the diagram, flows downward over the cascade terraces inserted. The vapor blows the liquid onto the perforated separating plates on which it flows down, where it again meets the vapor and, finally

flowing through the pipe reaches the next cascade terrace below.
The advantage of the cascade is that the meeting of vapor and
liquid is ensured by all distilling towers with any size diameters
and as a result the apparatus has a high effectiveness.

In using bubble-cap plates, especially in the case of towers
having large diameters, a separation of material might occur as
shown on the following diagram lowering the effectiveness of the
plates. There can be no such trouble when using cascade plates.

The vapors entering the tower must be condensed, other-
wise the entire quantity of vapor might leave at the top of the
tower and, lacking reflux, no fractionization appears. The
cooling of the tower is done by outside reflux; i.e. cool gaso-
line, or the cooled top cut from the tower is pumped back onto
the uppermost plate of the tower. The quantity of reflux is de-
termined by the heat balance already calculated.

As far as this calculation is concerned, let us examine
for example the heat balance if light gasoline, heavy gasoline,
kerosene, and gas oil are to be distilled from petroleum. The
lightest distillate, the light gasoline will leave at the top of
the tower; the other three fractions are obtained by tapping
the proper plates. Distillation of domestic petroleum is done
with a tower entrance temperature of 280 degrees, and the tower-
top temperature, if 25 percent light gasoline is wanted from the
petroleum, will be 110 degrees. (If desired a detailed calcu-
lation can be made for control). Heavy gasoline, kerosene, and
gas oil are drained from plates at a temperature of 140, 175, and
220 degrees respectively. (The plate temperature can also be
figured out and no mistake will be made if the temperature drop

between material input and tower-top temperatures are supposed to be linear.)

It was necessary to observe the above because, using these temperature data the reflux quantity needed for cooling can easily be calculated: the light gasoline fraction should be cooled from 280 degrees to 110 degrees; heavy gasoline from 280 degrees to 140 degrees; kerosene to 175 degrees, and gas oil to 220 degrees. Besides, the heavier distillates which have already changed to the vapor phase should also be cooled to about 240 degrees which is the temperature at the bottom of the tower. When using steam injection, the superheated steam should also be cooled up to 110 degrees Centigrade.

Taking into consideration that the specific heat of light gasoline is 0.52 and that of the heavier fractions 0.54 and 0.56, the heat quantity necessary for cooling can easily be figured out. The heat quantity calculated thus refers only to the sensible heat of the vapor phase. The heavy gasoline, kerosene, and gas oil must be condensed too; consequently, the loss of 69, 65, and 59 calories per kilogram of these products is also to be taken into consideration. Adding all these, we have the quantity of heat which must be removed by reflux, i.e. by heating the light gasoline which serves as reflux from 20 degrees to 110 degrees. The reflux thus calculated is a minimum quantity and more is always needed in practice. The reflux in general equals 2.5 to 4 times the top cut. By pressure-stabilization columns, to obtain products of the proper quality, a reflux equal to eight times the top cut is needed. When figuring the heat balance of the tower, the above facts should always be taken into consideration.

However, the tower may be cooled not only by leading back cooled products onto the upper plate but also for example, by pumping a cool material, tapped from a lower plate and cooled afterward, onto the plate between tapping heavy gasoline and kerosene.

Naturally in such cases only the sensible heat of the liquid used for reflux must be calculated. This procedure, however, has the advantage that hotter material is taken from the tower and which, therefore, can more economically be used for heat exchange. (In the case of the example mentioned, at top reflux the vapor is 110 degrees, at side reflux, however, a liquid of about 160 degrees can be produced between heavy gasoline and kerosene.)

As can be figured from the above in the tower, as a result of reintroducing the top reflux, the greatest quantity of vapor forms at the top of the tower. The quantity diminishes when using side reflux.

The vapor separation in the tower is shown in the following diagram: [See end of text].

The most important step in tower planning is the calculation of the tower diameter. This is done by considering the vapor velocity. Experience shows that, at a surface vapor velocity of 60 centimeters per second, the best separation of fractions can be made since, at this velocity, vapor and liquid show a strong desire to mix without foaming. This appears when, with lively mixing of liquid and vapor, small liquid drops of a diameter of 0.1 to 0.2 millimeters appear which are blown because of their greater vapor velocity onto the plate above, thereby lowering the

effectiveness of the plates. Experiments have proved this theory correct and it was shown that this form of foaming is negligible at a vapor velocity of 60 centimeters per second; at a velocity of 1.2 meters per second foaming equals 7 percent and at a velocity of 1.6 meters per second, it reaches 20.5 percent.

Naturally, foaming is influenced by the distance between plates. Foaming is smaller the greater the distance between plates. Alas, this means also that the height of the tower grows substantially, which again means that the equipment becomes more expensive. According to experiments, the optimum plate distance is 560 millimeters. At present towers working at nearly atmospheric pressure are generally built according to this measurement.

As it can be seen in the diagram, [end of text] the greatest quantity and velocity of the vapor appear at the top of the tower. Accordingly, the tower is to be planned so that the vapor velocity at top should not exceed 0.60 to 0.75 meters per second. For calculation of the mass velocity, the following formula can be used:

$$V = C \sqrt{d_2 (d_1 - d_2)}$$

where V equals velocity, d_1 is the density of the liquid at tower temperature, d_2 is the density of vapor also at tower temperature, and C is a constant, the value of which depends on the distance between plates, the vapor velocity, and the surface tension of the oil.

Apart from the top cut, the other distillations are taken out of the tower by the draining of the l-l plates. These products, of course, still contain other lighter distillates. The removal of

- 13 -

lighter distillates is done in the so-called vaporizing towers, by blowing in steam. In the vaporizing towers there are usually 4 to 5 bubble-cap plates. The diameter of the tower is figured according to above theories but it is substantially smaller than that of the main tower.

The vaporization of the bottom cut is done by plates placed under the raw material input and also by the use of steam.

The essence of the vaporizing towers consists of the lowering of the partial pressure, as a result of which the components with a lower boiling point become vaporized. By lowering the partial pressure, the boiling point also descends. If the molecules of oil vapor are called O and those of the steam G, the partial pressure of oil vapors is p_O and that of steam p_G, and the total pressure   ; then:

divided
$$\frac{O}{O + G} = \frac{pO}{\pi} \quad \text{and} \quad \frac{G}{O + G} = \frac{pG}{\pi}$$

but
$$\frac{G}{O} = \frac{pG}{pO} \quad \text{or} \quad G = O\left(\frac{pG}{pO}\right)$$

and
$$pG + pO = 1$$
$$G = O\,\frac{\pi - pO}{pO} = O\left(\frac{pG}{pO}\right)$$

Many researchers have found however, that steam more effectively lowers the boiling point, as established by Dalton's law. However, when making the calculation for planning, this does not make any considerable difference.

- 14 -

In practice the following vapor quantities are necessary for the vaporization of individual products:

Heavy gasoline 12-24 kilogram vapor per cubic meter of product

Kerosene 24-36 kilogram vapor per cubic meter of product

Gas oil 36-72 kilogram vapor per cubic meter of product

Heating oil 48-100 kilogram vapor per cubic meter of product

As it can be seen from these data, the vapor quantity may be changed within very wide limits. Naturally, when planning the tower, in the calculation of vapor velocities, the quantity of steam, and the heat balance, the heat content should always be taken into consideration.

The proper application of steam in the manufacturing process is extremely important. Therefore the constancy of vapor (steam) pressure is one of the basic conditions for the proper working of the tower. The separation of individual fractions could be regulated to a certain extent by the vapor quantity blown into the vaporizers but, in case of extreme vapor injection, the phenomena of foaming already mentioned strongly diminishes the sharpness of separation. Through improper handling, the excess of vapor often prevents the downward flow of condensed products from the plates and, at the top of the tower, all the material from the upper plates foam through without distillation.

The theory and practice of distillation have an extensive literature today; the professional magazines always publish the results of theoretical and practical progress. The purpose of the present article was to direct attention to those principles which are basic when planning and operating, and to show those

empirical data useful when planning a distilling tower, occasional

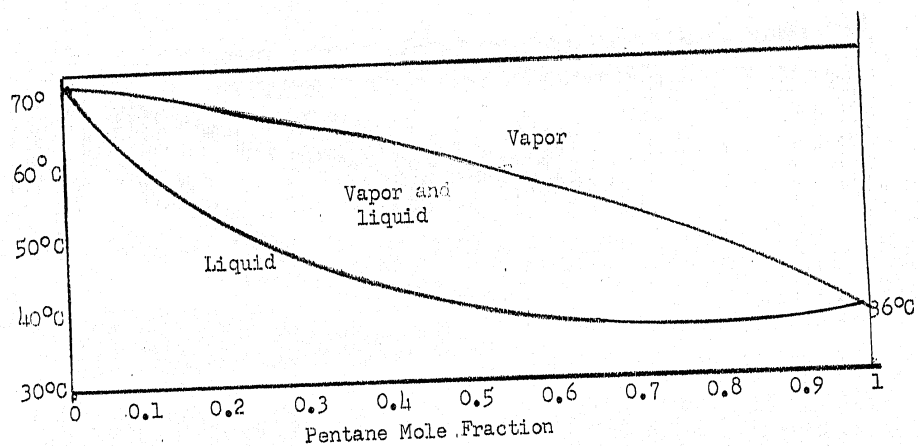checking of which is an absolute requirement for good manufacturing

work.

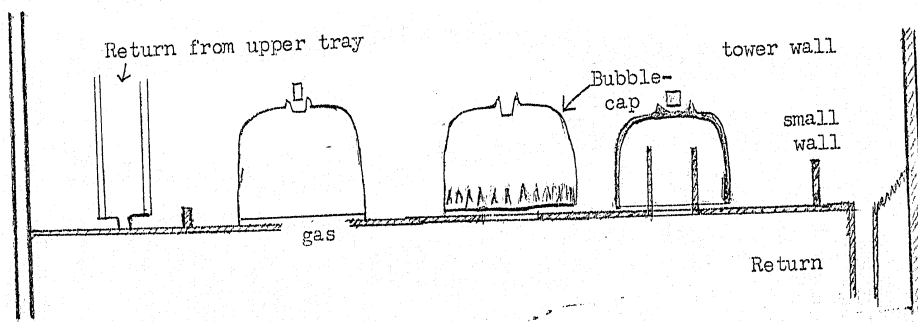Figure 1. Vapor-liquid equilibrium curve

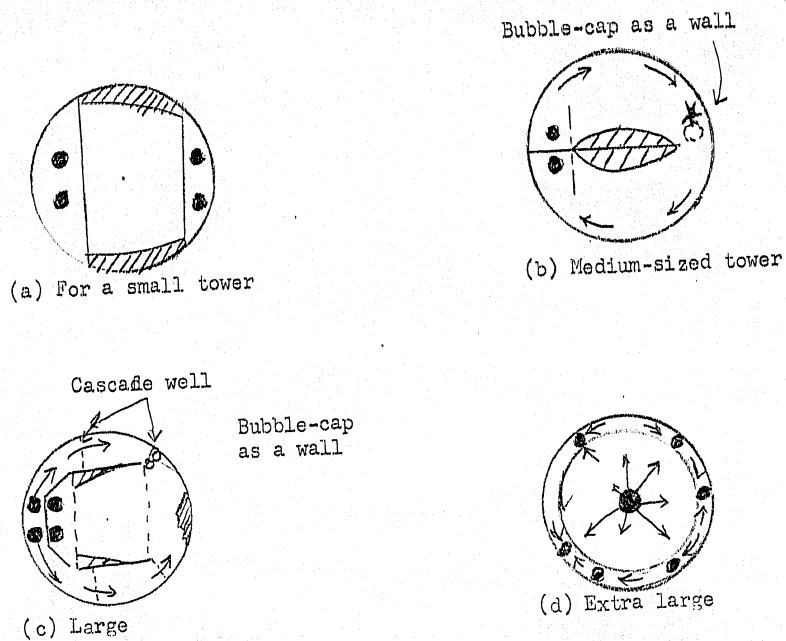Figure 2. Cross-section of bubble-caps and plate

(a) For a small tower

Bubble-cap as a wall

(b) Medium-sized tower

Cascade well

Bubble-cap
as a wall

(c) Large

(d) Extra large

Figure 3. Distillation plate reflux holes
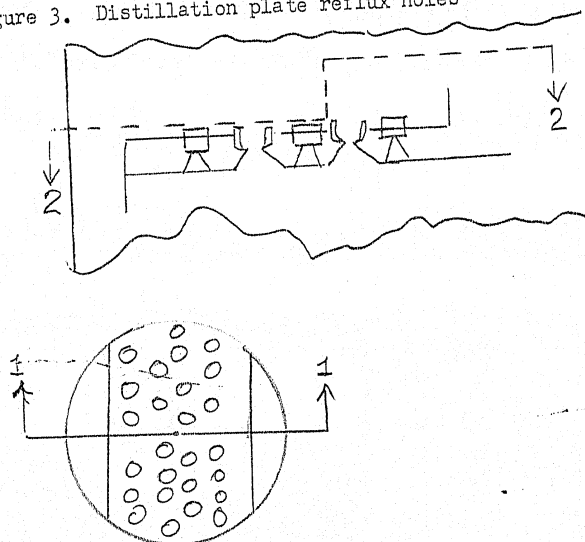
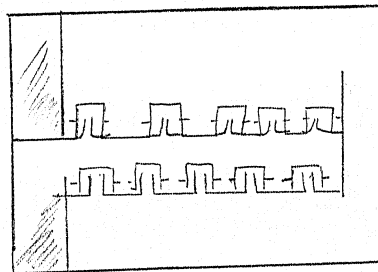Figure 4. Distilling Tower Plates
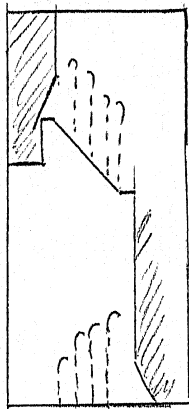
Figure 5. Cascade Insert
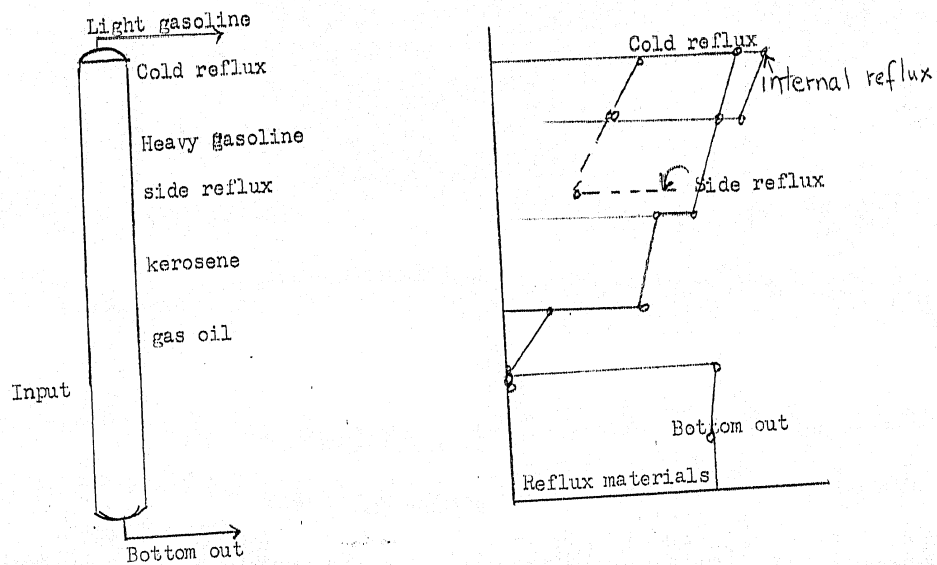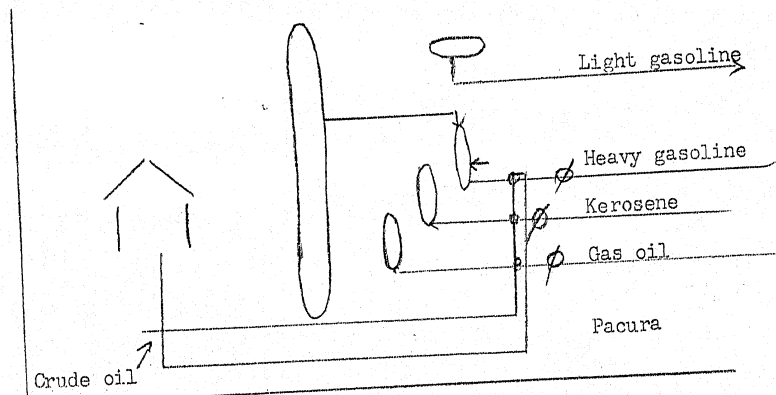

Figure 6. Bubble-cap Plate in Incorrect

Operation

Figure 7. Vapor Pattern of Distilling Tower

Figure 8. Distilling Tower